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METASTABLE METALLIC SUPERCONDUCTORS WITH HIGH TRANSITION TEMPER--ETC(U)
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by

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1. Final Report, Metastable Metallic Superconductors with High Transition Temperatures and Composite Superconductors

January 1, 1974 to December 31, 1977

AFOSR Contract No [illegible]620-74-C-0042

John R. Gavaler and Michael R. Daniel.

2. Abstract

Chemical vapor deposition (CVD) was found to be a practical method for preparing large quantities of Nb-Ge. The fabrication of Nb-Ge multifilamentary and tape conductors by this method was shown to be feasible. CVD Nb-Ge had critical temperatures and upper critical fields comparable to the sputtered material. Critical-current densities were lower. Second-phase doping was shown effective toward improving current-carrying capacity. A new Nb-Ge growth method (reactive sputtering of niobium in Argon/Germane) was demonstrated and its applicability for use in a magnetron sputtering system evaluated. A study of the effect of impurities (oxygen, nitrogen and silicon) on sputtered Nb-Ge films indicated that each of these impurities can stabilize the high-critical temperature metastable A15 phase. An investigation of the bronze diffusion process illustrated why A15 Nb-Ge cannot readily be made by this method. The formation of A15 Nb-Ga by bronze diffusion was achieved. The preparation of superconducting niobium and A15 structure Nb-Sn was accomplished by liquid sodium reduction of the halides. The anomalously high upper critical field of sputtered NbN film was shown to be due to a surface critical field.

3. Original Objectives

The objectives of this program can be summarized as follows:

1. Prepare high-transition temperature metastable metallic superconductors using low-energy sputtering and other novel methods.
2. Characterize superconducting properties.
3. Characterize chemical and metallurgical properties.
4. Characterize mechanical properties.
5. Study the interrelationship of physical, chemical and metallurgical properties with a view to maximizing critical temperature.
6. Investigate methods by which superconductors could be fabricated into technologically-useful configurations.
7. Exploit the special properties of these materials for superconducting device concepts.

High-temperature superconducting materials are needed for application in airborne multimegawatt power generation and conditioning systems. Research directed toward the synthesis and characterization of high-transition temperature (T_c) metallic compounds can eventually provide a greater margin of design efficiency and operational reliability than obtained with presently-available superconductors. In the previous AFOSR-Westinghouse program (Contract No. F44620-71-C-0045), the A15 structure intermetallic compound, Nb_3Ge, was prepared. This compound, with a T_c of $\sim$ 23 K, is the highest temperature superconductor now known. It was synthesized in a metastable form by a specially-developed, low-energy

sputtering process. Because of this result, the present program was focused on high-T_c metastable compounds and composites with particular emphasis placed on Nb_3Ge. Some of the important results of the program are briefly summarized. These results are categorized according to the above-listed objectives. More detailed discussion can be found in the publications listed in Section 5.

4. Accomplishments

4.1 Superconductor Preparation

In addition to the original low-energy sputtering process used to prepare Nb_3Ge, other preparation methods investigated were: reactive sputtering, chemical vapor deposition, "bronze" diffusion and liquid-sodium reduction. A method for preparing Nb_3Ge by the reactive sputtering of niobium in argone-germane (GeH_4) mixtures was discovered. The attempt was made to incorporate this method into a magnetron-sputtering system. Magnetron sputtering, although a low-energy process, has been shown in work at other laboratories to be able to deposit high-T_c Nb_3Ge at high rates. Results of this effort showed that the reactive sputtering of Nb_3Ge in a magnetron system was feasible however the deposition rates were lower than expected. These low deposition rates were attributed to the generation of hydrogen from the decomposition of GeH_4.

Chemical vapor deposition, another high-deposition rate method, was also investigated for preparing Nb_3Ge in practical quantities. With this method Nb_3Ge films were deposited at high rates which had T_c's the same as those achieved in sputtered films.

The possible formation of A15 structure compounds, including Nb_3Sn, Nb_3Ga and Nb_3Ge by "bronze" source diffusion was studied. Similar to previous results Nb_3Sn was formed readily by this method. Nb_3Ga was formed only after the bronze was modified by replacement of copper with silver. Nb_3Ge could not be prepared.

An effort was made to develop a method for synthesizing metastable superconductors by the liquid sodium reduction of the constituent oxides or halides. Experimental work in this effort was primarily concentrated on the Nb-Ge system. The attempted reduction of the Nb- and Ge-oxides were unsuccessful with only partial reduction occurring.

However complete reduction of Nb, Ge, and Sn-halides was achieved. Niobium particles, about 300 Å in diameter, obtained by this method were found to be superconducting with T_c's of $\sim$ 8 K.

To demonstrate the efficacy of this method for preparing A15 structure compounds, a mixture of Nb and Sn-halides was reduced to form A15 Nb_3Sn. Efforts to prepare Nb_3Ge and other of the more unstable A15 superconductors by this method, are being continued.

4.2 <u>Characterization of Superconducting Properties</u>

Extensive studies of superconducting properties other than T_c were made primarily on sputtered and CVD films of Nb_3Ge. Chemically vapor deposited Nb_3Ge was found to have critical temperatures and upper critical fields the same or close to that achieved in sputtered material. Critical current densities were about an order of magnitude lower. Since the sputtered films had much smaller grain sizes, it was concluded that the higher critical currents in the sputtered films were due to the flux pinning at the grain boundaries. Improved flux pinning in CVD films was obtained by second-phase doping either by the use of a dopant gas (N_2 or C_2H_6) or by changing the Nb/Ge ratio sufficiently to produce a two-phase (A15 and σ-phase) material. By the inclusion of an appropriate amount of the second-phase, higher critical-current densities similar to those of sputtered material were obtained, particularly at lower fields. Comparable high-current densities were also obtained in CVD material when prepared with a fine-grained microstructure.

Sputtered NbN film which has a column-void type microstructure has been shown to possess an anomalously high upper critical field in the direction normal to the plane of the film. A theoretical proposal, which was experimentally verified, removed this anomaly by showing that this critical field is H_{c3}, the field due to superconductivity at the surface of the vertical columns in the films.

4.3 <u>Characterization of Chemical and Metallurgical Properties</u>

Attempts to obtain the optimum properties in a Nb_3Ge and other A15 structure materials included investigation of their phase equilibria,

composition, stoichiometry, crystallographic structure and microstructure. Among these data, the following results are considered particularly noteworthy.

A new tetragonal phase (T2) was discovered in the Nb-Ge system. This phase is believed to be an impurity-stabilized phase with the impurity probably being chlorine.

The problem of Nb_3Ge Transmission Electron Microscope (TEM) sample preparation was solved and thus thin foils suitable for electron microscopy were successfully prepared. Hence for the first time the microstructures of high-T_c Nb_3Ge films could be studied by TEM. Furthermore, selected area electron diffraction and a high resolution dark-field technique were used for the nonambiguous identification of Nb-Ge σ-phase particles dispersed in Nb_3Ge.

Phase equilibria studies were conducted in the ternary systems, Nb-Sn-Cu, Nb-Ga-Cu and Nb-Ge-Cu and also in analogous systems where Ag and Ni were substituted for Cu. This study showed that the possible diffusion paths from the Cu-rich bronze compositions were connected directly across the Nb_3Sn (A15) composition and thence to the pure Nb explaining why Nb_3Sn can readily be formed by the "bronze" technique. In the Nb-Ge-Cu and the Nb-Ga-Cu systems there was no evidence of any direct paths to the A15 phase field. Substituting Ag for Cu showed that this was a feasible method for promoting A15 layer formation as indicated by the successful growth of A15 structure Nb_3Ga in the Nb-Ga-Ag system. However, no A15 Nb_3Ge was obtained in any of the systems studied.

4.4 Characterization of Mechanical Properties

One of the most important properties that helps determine the practicality of a superconductor is its resistance to critical-current degradation from the effect of strain. To evaulate the effect of strain on Nb_3Ge at cryogenic temperatures a collaboration was initiated with the National Bureau of Standards in Boulder, Colorado. Films were provided to J. Ekins of NBS and the pertinent measurements are being made.

4.5 Interrelationships of Physical, Chemical and Metallurgical Properties with T_c

The preponderance of data obtained in this program supports the view that 3/1 stoichiometry is required to obtain the highest T_c in the Nb-Ge system.

Strong circumstantial evidence was also obtained which indicated the importance of impurities (such as oxygen, nitrogen and silicon) on the stabilization of this high-T_c stoichiometric Nb_3Ge phase. The mechanism by which this occurs is still an open question. However, the experimental data, including structural and chemical analysis of very thin (< 1000 Å) films, indicated that the impurities influence the initial nucleation process of the Nb_3Ge phase, and thereafter are not required for the continued growth of this phase.

4.6 Methods for Fabricating Technologically-Useful Configurations

Modification of "bronze" diffusion method by substituting Ag for Cu in Nb-Ga-Cu system permitted the formation of Nb_3Ga. This indicates the possibility that multifilamentary Nb_3Ge wire may be accessible by this method.

Another method for forming multifilamentary-type wire was carried out by chemically vapor depositing Nb_3Ge on a 200 fiber Al_2O_3 yarn. The individual fiber thickness was 20 μm. Simultaneous coating of individual fibers was achieved. The T_c's of these fibers ranged betweem 21 and 22 K. This result demonstrated the feasibility of fabricating this type Nb_3Ge multifilamentary conductor since there are presently-available methods by which such fibers could be incorporated into an appropriate metal matrix to form the desired composite conductor.

Niobium-germanium was also successfully deposited on long lengths of metal tape and then covered with a layer of copper in a typical configuration used for certain types of solenoid windings where a multifilamentary wire is not mandatory.

4.7 Superconducting Device Concepts

Samples of Nb_3Ge on sapphire and on metallic tapes were prepared for use in other programs to evaluate the potential of Nb_3Ge for Josephson devices and for very high-field magnets ($\gtrsim$ 200 kG).

5. Publications

1. "Chemical Vapor Deposition of Superconducting Nb_3Ge Having High Transition Temperatures," A. I. Braginski and G. W. Roland, Appl. Phys. Letters, December 15, 1974.

2. "Superconductivity in Nb_3Ge," J. R. Gavaler, M. A. Janocko, A. I. Braginski, and G. W. Roland, IEEE Transactions on Magnetics, MAG-11, #2, March 1975.

3. "Properties of High T_c Thin Films," J. R. Gavaler, J. Vac. Sci. Technol. 12, 103 (1975).

4. "Mass-Spectrographic Analysis of High-T_c Nb-Ge Sputtered Films," A. T. Santhanam and J. R. Gavaler, J. Appl. Phys. 46, 3633 (August 1975).

5. "Oxygen Distribution in Sputtered Nb-Ge Films," J. R. Gavaler, J. W. Miller, and B. R. Appleton, Appl. Phys. Letts. 28, 237 (February 15, 1976).

6. "Preparation of Nb_3Ge by Chemical Vapor Deposition," A. I. Braginski, G. W. Roland, and Michael R. Daniel, Appl. Polymer Symposium No. 29, 93-104 (1976).

7. "Critical Current Density and Flux Pinning in Nb_3Ge," A. I. Braginski, Michael R. Daniel, and G. W. Roland, AIP Conference Proceedings No. 34, p. 78 (1976).

8. "Critical Current Determination from the Magnetization of Type II Superconducting Disc Samples," Michael R. Daniel and M. Ashkin, Cryogenics, 16, 610 (1976).

9. "Projected Upper Limits to the Critical Current Density in Nb_3Ge, Nb_3Sn and V_3Ga," Michael R. Daniel, Cryogenics, 16, 727 (1976).

10. "Phase Relations and Diffusion Layer Formation in the Systems Cu-Nb-Sn and Cu-Nb-Ge," R. H. Hopkins, G. W. Roland, and Michael R. Daniel, Metallurgical Transactions, A, 8A, 91 (1977).

11. "Temperature Dependence of the Critical Current Density in Nb_3Ge," Michael R. Daniel, A. I. Braginski, G. W. Roland, J. R. Gavaler, L. R. Newkirk, and R. J. Bartlett, J. Appl. Phys., 48, 1293 (1977).

12. "Impurity Stabilization of Nb_3Ge," J. R. Gavaler, Superconductivity in d- and f-Band Metals, p. 421, Ed. D. H. Douglas, Plenum Press, New York (1977).

13. "Progress Toward a Practical Nb-Ge Conductor," A. I. Braginski, J. R. Gavaler, G. W. Roland, Michael R. Daniel, M. A. Janocko, and A. T. Santhanam, IEEE Trans. on Magnetics, MAG-13, 300 (1977).

14. "Revealing the Microstructure of Nb_3Ge Superconducting Films by Transmission Electron Microscopy," A. T. Santhanam and P. M. Yuzawich, J. Mater. Sci., 12, 1161 (1977).

15. "Identification of the Tetragonal Nb_5Ge_3 Phase in Two-Phase Nb-Ge Superconducting Films by Electron Diffraction," A. T. Santhanam, J. Appl. Phys., 48 No. 8, 3347 (1977).

16. "Stability of Nb-Based High-T_c A15 Compounds," M. Ashkin and J. R. Gavaler, Accepted for publication in J. Low Temp. Phys.

17. "The Upper Critical Field of NbN Films," M. Ashkin and J. R. Gavaler, Accepted for publication in J. Appl. Phys.

18. "Impurity Doping of CVD Nb_3Ge and Its Effect on Critical-Current Density," A. I. Braginski, G. W. Roland, M. R. Daniel, A. T. Santhanam and K. W. Guardipee, Accepted for publication in J. Appl. Phys.

19. "Nb_3Ge as a Potential Candidate Material for 15 to 25 T Magnets," M. R. Daniel, A. I. Braginski, G. W. Roland, J. R. Gavaler and A. T. Santhanam, Accepted for publication in the Proc. CEC-ICMC Conf., Boulder, CO (August 1977).

20. "Phase Relations and A15 Phase Diffusion Layer Formation in the System Ag-Nb-Ga," R. H. Hopkins and A. M. Stewart, Submitted for publication in Metallurgical Trans.

In Preparation

1. "Impurity-Induced Nucleation of the High-T_c A15 Nb-Ge Phase."
2. "Preparation of Superconductor by the liquid Sodium Reduction of the Constituent Halides."
3. "Microstructure and Critical-Current Density in Nb_3Ge."
4. "High-Field Properties of Nb_3Ge."
5. "Reactive Magnetron Sputtering of Nb_3Ge."

6. Personnel Contributing to Program

J. R. Gavaler	M. A. Janocko
M. R. Daniel	M. Ashkin
A. I. Braginski	A. T. Santhanam
G. W. Roland	R. G. Charles
R. H. Hopkins	K. W. Guardipee
D. W. Deis	R. D. Blaugher
C. K. Jones	R. Mazelsky
J. H. Parker, Jr.	M. P. Mathur

7. Coupling Activities

1. "High Critical Temperatures A15 Structure Superconductors," by J. R. Gavaler, Invited talk at Conference on "The Search for Higher Critical Temperature Superconductors," Kfar Giladi, Israel (June 1974).

2. "Superconductivity in Nb_3Ge," by J. R. Gavaler, Invited talk at Applied Superconductivity Conference," Oakbrook, Illinois (September 1974).

3. "The Properties of Sputtered High T_c Thin Films," Invited talk at the American Vacuum Society Meeting, Anaheim, California (October 1974).

4. "Type II Superconducting Thin Films," by J. R. Gavaler, Invited talk at the Annual Meeting of the American Physical Society, Anaheim, California (January 1975).

5. "Properties of Nb_3Ge Films Prepared by CVD," by A. I. Braginski, Seminar at Brookhaven National Laboratory, April 17, 1975.

6. "Chemical Vapor Deposition of Nb_3Ge," by A. I. Braginski, Contributed paper at the International Cryogenic Material and Cryogenic Engineering Conference held at Kingston, Ontario, July 1975.

7. "Preparation of Nb_3Ge by CVD," by G. W. Roland, Invited talk at American Chemical Society Meeting, Chicago, August 20, 1975.

8. "Effect of Oxygen on T_c of Nb-Ge Sputtered Films," Seminar by J. R. Gavaler, University of Giessen, West Germany, August 22, 1975.

9. "Properties of Nb_3Ge Prepared by CVD," by A. I. Braginski, Seminar at University of Southern California on September 8, 1975 and on September 18, 1975 at Case Western Reserve University.

10. "Recent Progress in CVD Processed Nb Ge," by A. I. Braginski on March 24, 1976, Seminar at Brookhaven National Laboratory.

11. "Projected Upper Limits to the Critical Current Density of Nb_3Ge, Nb_3Sn, and V_3Ga," by Michael R. Daniel, Invited colloquium at NASA-Lewis, April 1976.

12. Discussion on superconducting material research among J. Livingston (General Electric), J. R. Gavaler, A. I. Braginski, and M. Ashkin at Rochester, New York on April 30, 1976.

13. "Impurity Stabilization of Nb_3Ge," by J. R. Gavaler, Invited talk at 2nd Rochester Conference on Superconductivity in D- and F-Band Metals (May 1, 1976).

14. Discussion among C. Rosner, P. Swartz, and M. S. Walker (all of Intermagnetics General Corporation) and A. I. Braginski on possible collaborations in area of the development of Nb-Ge conductor at Guilderland, New York, May 26, 1976.

15. Discussion among E. Adam, E. Gregory (both of AIRCO Central Research Laboratories) and J. R. Gavaler on possible collaborations in area of magnetron sputtering of Nb_3Ge, at Murray Hill, New Jersey, May 1976.

16. "Critical Current Density and Flux Pinning in Nb_3Ge," by A. I. Braginski, Contributed talk at the Joint MMM-Intermag Conf., June 1976.

17. "Metastable Crystalline Superconductors," by J. R. Gavaler, Invited talk at Gordon Research Conference on Amorphous Metals, Plymouth, New Hampshire, July 1976.

18. "Progress Toward a Practical Nb-Ge Conductor," by A. I. Braginski, Invited talk at the Applied Superconductivity Conference, Palo Alto, California, August 1976.

19. "Phase Relations and Diffusion Layer Formation in the Systems Cu-Nb-Sn and Cu-Nb-Ge," by Michael R. Daniel, Contributed talk at the TMS-AIME Fall Meeting in Niagara Falls, New York, September 1976.

20. "Preparation and Properties of Thin Film Type II Superconductors," by J. R. Gavaler, Invited talk at the TMS-AIME Fall Meeting in Niagara Falls, New York, September 1976.

21. "Critical Current Density in Impurity-Doped Nb_3Ge," by A. I. Braginski, Contributed talk at the TMS-AIME Symp. on Superconducting Materials and Applications, Niagara Falls, New York, September 21, 1976.

22. "Fabrication of Nb_3Ge Tapes by CVD," by A. I. Braginski, Contributed talk at ASM Conference on Manufacture of Superconducting Materials, Port Chester, November 10, 1976.

23. "High-T_c Studies at Westinghouse," by A. I. Braginski, Invited seminar at MIT-National Magnet Laboratory, March 14, 1977.

24. "Symposium on Superconductivity," Organized and chaired by J.R. Gavaler, Speakers included T. H. Geballe (Stanford University), A. R. Sweedler (Brookhaven National Laboratory), J. R. Rowell (Bell Telephone Labs), and J. Cheng (California Tech.), March Meeting of the APS, San Diego, California, March 1977.

25. Workshop on "Superconducting Materials," Sponsored by ERDA, Participants: R. Hein (Naval Research Laboratory), M. Suenaga (Brookhaven National Laboratory), G. Webb (University of California), M. Beasley (Stanford University), A. Giorgi (Los Alamos Scientific Laboratory), C. Henning (Energy Research and Development Administration), R. Dynes (Bell Telephone Labs), and J. R. Gavaler at Brookhaven National Laboratory, April 1977.

26. "Phase Relations and Diffusion Layer Synthesis in Candidate Superconducting Systems," by R. H. Hopkins, Invited talk at the University of Pittsburgh, Pittsburgh, Pennsylvania, May 1977.

27. Participation in the Discussion Conference on "Irradiation Effects in Superconductors," Argonne, Illinois, June 1977, M. Ashkin and A. I. Braginski.

28. "Nb Ge as a Potential Candidate Material for 15 to 25 T Magnets," by A. I. Braginski, Contributed talk at CEC-ICMC Conf., Boulder, CO (August 1977).

29. "Nb Ge Superconductors for Magnets," by A. I. Braginski, Seminar at National Science Foundation, Washington, DC (October 28, 1977).

8. Patents and Inventions

1. Patent Disclosure on Making Multifilamentary Nb_3Ge using CVD.
2. Patent Disclosure on the Preparation of Multifilamentary Wire from Brittle Type II Superconductors using Liquid Sodium Solution Growth Methods.
3. Patent Disclosure on the Reactive Magnetron Sputtering as a Possible High Rate Sputtering Method for Preparing Nb_3Ge.

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Chemical vapor deposition (CVD) was found to be a practical method for preparing large quantities of Nb-Ge. The fabrication of Nb-Ge multifilamentary and tape conductors by this method was shown to be feasible. CVD Nb-Ge had critical temperatures and upper critical fields comparable to the sputtered material. Critical-current densities were lower. Second-phase doping was shown effective toward improving current-carrying capacity. A new Nb-Ge growth method (reactive sputtering of niobium in Argon/Germane) was demon-

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demonstrated and its applicability for use in a magnetron sputtering system evaluated. A study of the effect of impurities (oxygen, nitrogen and silicon) on sputtered Nb-Ge films indicated that each of these impurities can stabilize the high-critical temperature metastable A15 phase. An investigation of the bronze diffusion process illustrated why A15 Nb-Ge cannot readily be made by this method. The formation of A15 Nb-Ga by bronze diffusion was achieved. The preparation of superconducting niobium and A15 structure Nb-Sn was accomplished by liquid sodium reduction of the halides. The anomalously high upper critical field of sputtered NbN film was shown to be due to a surface critical field.